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“पुराने को छोड़ नये के तरफ”

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“Step Out From the Old to the New”

IS 505 (1995): Light kaolin [CHD 1: Inorganic Chemicals]



“ज्ञान से एक नये भारत का निर्माण”

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Bhartrhari—Nitiśatakam

“Knowledge is such a treasure which cannot be stolen”

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भारतीय मानक

हल्का केओलिन — विशिष्ट

(तीसरा पुनरीक्षण)

Indian Standard

LIGHT KAOLIN — SPECIFICATION

(*Third Revision*)

UDC 666.321

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BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

FOREWORD

This Indian Standard (Third Revision) was adopted by the Bureau of Indian Standards, after the draft finalized by the General Inorganic Chemicals Sectional Committee had been approved by the Chemical Division Council.

This standard was first published in 1958 under the title China clay for rubber industry. It was revised in 1968 amalgamating it with IS 1092 : 1957 Specification for China clay for textile and paper industries under the title light kaolin. The requirements of insecticide and pharmaceutical industries were added in the first revision.

In the second revision brought out in 1978 two different grades of the material for paper industry were prescribed. The grade meant for the pharmaceutical industry was excluded as it is covered in the Indian Pharmacopoeia. Changes were made in the requirements for coarse particles, particles larger than 10 microns and particles smaller than 2 microns in view of new methods of test prescribed for these characteristics. The requirements of grit chloride, carbonate and sedimentation volume were deleted.

In this revision, only two grades of light kaolin have been prescribed. The grade 1 material is for use as coating clays in paper industries whereas the Grade 2 material is suitable for use as filler in rubber, paper, textiles industries, etc. Various requirements for both grades of light kaolin have been modified.

The composition of the committee responsible for formulation of the standard is given at Annex C.

For the purpose of deciding whether a particular requirement of this standard is complied with, the final value observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

Indian Standard

LIGHT KAOLIN — SPECIFICATION

(Third Revision)

1 SCOPE

This standard prescribes the requirements and the methods of sampling and test for light kaolin.

2 REFERENCES

The Indian Standards listed below are necessary adjunct to this standard:

IS No.	Title
264 : 1976	Nitric acid (<i>second revision</i>)
265 : 1993	Hydrochloric acid (<i>fourth revision</i>)
266 : 1993	Sulphuric acid (<i>third revision</i>)
296 : 1986	Sodium carbonate (<i>third revision</i>)
323 : 1959	Rectified spirit (<i>revised</i>)
460 : 1985	Test sieves (<i>third revision</i>)
1070 : 1992	Reagent grade water (<i>third revision</i>)
2088 : 1983	Methods for determination of arsenic (<i>second revision</i>)
3205 : 1984	Precipitated barium carbonate, technical (<i>first revision</i>)
4161 : 1967	Nessler cylinders
4905 : 1968	Methods for random sampling

3 GRADES

There shall be following two grades of the material:

- Grade 1 suitable for paper industry, and
- Grade 2 suitable for use as general filler.

4 REQUIREMENTS

4.1 Description

The material shall be the beneficiated natural mineral powder consisting essentially of hydrated aluminium silicate. It shall be dry and free from extraneous impurities and grit.

4.1.1 For use in textile and paper industries, the colour of the material shall be white, while for use in rubber and insecticides the colour may be white or cream.

4.1.2 The material shall not contain any bluing or whitening substances.

4.2 The material shall also comply with the requirements laid down in Table 1, when tested in accordance with the methods prescribed in Annex A. Reference to the relevant clauses of Annex A are given in col 7 of the table.

5 PACKING AND MARKING

5.1 Packing

The material shall be packed in paper bags or hessian bags with a suitable liner.

5.1.1 For special requirements, the material shall be supplied in wagons if agreed to between the purchaser and the supplier.

5.2 Marking

Each container shall be marked legibly and indelibly with the following information:

- a) Name and grade of the material,
- b) Indication of the source of manufacture,
- c) Gross and net mass,
- d) Date of packing, and
- e) Batch number.

5.2.1 BIS Certification Marking

The bags may also be marked with the Standard Mark.

5.2.1.1 The use of the Standard Mark is governed by the provisions of the *Bureau of Indian Standards Act, 1986* and the Rules and Regulations made thereunder. The details of conditions under which the licence for the use of the Standard Mark may be granted to manufacturers or producers may be obtained from the Bureau of Indian Standards.

6 SAMPLING

The method of drawing representative samples of the material from a lot, the number of tests to be performed and the manner of determining the criteria for conformity of the material to the requirements of this specification shall be prescribed in Annex B.

Table 1 Requirements for Light Kaolin
(Clause 4.2)

Sl No.	Characteristics	Requirements for		Method of Test, Ref to Cl No. in Annex A
		Grade 1	Grade 2	
(1)	(2)	(3)	(4)	(5)
i)	Coarse particles or grit represented as residue on 53-micron IS sieve (see IS 460 : 1985), percent by mass, <i>Max</i>	0.8	1.0	A-2
ii)	Particles larger than 10 microns in diameter, percent by mass, <i>Max</i>	5.0	15.0	A-3
iii)	Particles smaller than 10 microns in diameter percent by mass, <i>Min</i>	75	60	A-4
iv)	Relative density at 27/27°C	2.5 to 2.9	2.5 to 2.9	A-5
v)	Loss on drying, percent by mass, <i>Max</i>			
	Lump	10	10	A-6
	Powder	2	2	
vi)	Loss on ignition, percent by mass, <i>Max</i>	14.0 to 15.5	14.0 to 15.5	A-7
vii)	Matter soluble in water, percent by mass, <i>Max</i>	—	0.5	A-8
viii)	Matter soluble in hydrochloric acid, percent by mass, <i>Max</i>	0.5 to 1.0	1.5 to 2.5	A-9
ix)	Copper (as CuO), percent by mass, <i>Max</i>	—	0.007	A-10
x)	Arsenic (as As ₂ O ₃), parts per million, <i>Max</i>	—	10	A-11
xi)	Iron (as Fe ₂ O ₃), percent by mass, <i>Max</i>	0.6	0.75	A-12
xii)	Manganese (as MnO), percent by mass, <i>Max</i>	—	0.013	A-13
xiii)	pH value (of aqueous extract)	4.5 to 7.5	4.5 to 7.5	A-14
xiv)	Oil absorption, ml per 100 g, <i>Min</i>	—	50	A-15
xv)	Colour reflectance to blue light of wave length 5 040 Å, percent	80 to 85	As agreed to between the purchaser and the supplier	A-16

ANNEX A

(Clause 4.2 and Table 1)

METHODS OF TEST FOR LIGHT KAOLIN

A-1 QUALITY OF REAGENTS

Unless specified otherwise, pure chemicals and reagent grade water (see IS 1070 : 1992) shall be employed in tests.

NOTE—'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

A-2 COARSE PARTICLES OR GRIT REPRESENTED AS RESIDUE ON 53-MICRON IS SIEVE

A-2.1 Reagents

A-2.1.1 Tetrasodium Pyrophosphate Solution — approximately 1 percent (m/v) (on anhydrous basis).

A-2.2 Procedure

Weigh about 200 g of the material and add it to about 1 litre of water in a glass or plastic vessel which is capable of being closed with a cap or bung. The vessel shall be of such a capacity that it is more than about two-thirds full. Add 50 ml of tetrasodium pyrophosphate solution (this makes the wetting and subsequently dispersal and sieving easier). Close it, shake briefly and allow to stand for about 30 min to allow the material to wet thoroughly.

A-2.2.1 Mount the 53-micron IS sieve over a sink and pour the slurry carefully from the vessel into the sieve. The passage through the

sieve is assisted by impingement of a stream of water directed by hand from a flexible tube attached to the water supply. The stream also serves to wash out the last traces of solids from the vessel into the sieve. Wash the residue on the sieve until the washings are clear and contain no solid particles, and then transfer by washing into a porcelain dish. Evaporate the water and transfer the dry residue to a watch-glass by brushing with a fine bristles brush.

A-2.3 Calculation

$$\text{Coarse particles, percent by mass} = 100 \times \frac{M_1}{M_2}$$

where

M_1 = mass in g of the residue obtained, and

M_2 = mass in g of the material taken for the test.

A-3 PARTICLES LARGER THAN 10 MICRONS IN DIAMETER

A-3.0 Outline of the Method

The method prescribed is the beaker sedimentation method and is useful for measurement in the range 4 to 40 microns. It is based on Stokes law which defines the constant velocity reached by a sphere falling without turbulence, through a viscous fluid:

$$V = \frac{gD^2 (\Delta d)}{18\eta}$$

where

V = sedimentation velocity in cm/s,

g = acceleration due to gravity in cm/sec²,

D = diameter of sphere in cm,

Δd = difference in density between the sphere and the fluid,

η = viscosity of the fluid in poises.

(For china clay and water Δd is 1.6.5 g/ml).

η is temperature dependent so that if temperature is kept constant, η , Δd , and g are constants and V is proportional to the square of the diameter of the particles.

Using this relationship it is possible to predict the depth of particles, starting in the free surface of the suspension, after a certain time has elapsed, clay particles are not spherical and particles sizes are expressed as equivalent spherical diameters or stokes diameters. For this test it is important that two conditions are met: (a) The clay shall be completely dispersed and deflocculated, and (b) The concentration of the solids in the suspension should not exceed 5 percent by mass.

A-3.1 Reagents

A-3.1.1 Deflocculant Solution

Prepared by mixing 10 g of anhydrous sodium carbonate and 5 g of sodium hexametaphosphate in 1 litre of water.

A-3.2 Procedure

Take a glass cylinder having the approximately dimensions 5 cm internal diameter and height of 30 cm, and a painted or inscribed meniscus mark at a height of 20 cm above the internal base. Place into this cylinder 20 ml of the deflocculant solution and 20.0 g of dry material. Add water until the meniscus is at the mark. Set aside the cylinder for about one hour after which close it with a rubber bung and shake vigorously in order to disperse the clay. Place the cylinder on the bench and note the time. After 40 minutes decant the liquid carefully from the residue (a better alternative is to remove the liquid with a baffled siphon tube connected to a water jet vacuum pump; this technique leaves the residue and about 1 cm of liquid above it undisturbed). Fill the cylinder again to the mark with water and 5 ml of the deflocculant solution, shake to resuspend the particles and after a further 40 minutes remove the liquid again. Repeat this process twice to make a total of four sedimentations and decantations.

A-3.2.1 Wash out the final residue with water into a porcelain dish and dry. Transfer the dry residue with a brush to a watch glass and weigh. This mass in g multiplied by 5 gives the percentage by mass of particles coarser than 10 microns.

NOTE — Strictly these conditions apply to a temperature of 17.5°C but for coarser particles, temperature fluctuations give rise to comparatively small errors and thermostatic water baths are not essential. Corrections for ambient temperature are given below:

Temperature (°C)	Time at 20 cm depth (minutes)
14	43.75
16	41.5
19	38.5
20	37.5
22	35.75

A-4 PARTICLES SMALLER THAN 2 MICRONS IN DIAMETER

A-4.0 Outline of the Method

The method prescribed is the pipette Andreasen method, and is useful for measurements in the range 0.5 to 4 microns. It is based on Stokes law and the principle is the same as described for the beaker method in A-3.0.

A-4.1 Apparatus

The apparatus consists of a thermostatic water bath, glass cylinders, tared glass sample dishes and a special pipette. The latter is of 20 ml capacity and is mounted in a holder adjustable in height and in two directions in a horizontal plane. Thus a number of cylinders may be sampled in turn without disturbing their contents. The pipette holder also carries a probe which, when brought into the meniscus of suspensions, would dictate the depth of the pipette tip below the meniscus. Large vertical movements of the pipette are achieved by means of a sliding clamp and fine movements by means of a rack and pinion gear. The pipette has two vacuum services from a single pump which enable samples to be taken either rapidly or slowly. For slow sampling, the filling time shall not be less than 15 seconds.

A-4.2 Reagents

A-4.2.1 Deflocculant Solution

Prepared by mixing 10 g of the anhydrous sodium carbonate and 5 g of sodium hexametaphosphate in 1 litre of water.

A-4.3 Procedure

Take about 5 g of the material which need not be dried or weighed accurately, and add to 5 ml of the deflocculant solution in a cylinder of diameter 5 to 6 cm. Add water to a total volume of about 250 ml and place the cylinder in a water bath whose liquid depth is such that no part of the suspension column is exposed.

A-4.3.1 Allow a period of one hour to elapse to equalize temperatures and to allow the clay to wet thoroughly. Remove the cylinder, close with a bung and shake vigorously to disperse the soaked clay lumps. Replace the cylinder in the bath and immediately sample with the pipette regardless of pipette depth. Discharge the 20 ml sample into a tared glass dish which is placed on a water bath or suitable hot plate and evaporate its contents to dryness without boiling. The mass of the clay from the sample is found to the nearest milligram (*A*).

A-4.3.2 If the temperature of the bath is 24°C, withdraw a further sample slowly 3 hours 39 minutes after the first sample and for this, the pipette is lowered carefully into the liquid to avoid disturbing it. By means of tube probe index, take the sample from a depth of 5.0 cm below the free surface. The mass of solids in this second sample is determined as before (*B*).

A-4.4 Calculation

Mass *B*, expressed as a percentage of *A*, gives the percentage by mass of particles finer than

2 microns. A small correction shall be applied because the soluble salts of the deflocculant solution are counted erroneously as clay. An empirical correction *C* shall be applied by running a blank determination on a cylinder made up as usual but containing no clay. The mass of dissolved solids so obtained can be subtracted from *A* and *B*.

$$\text{Particle smaller than 2 microns, percent by mass} = \frac{B - C}{A - C} \times 100$$

NOTE — The temperature of 24°C has been chosen in the above determination because the control of temperature is facilitated if the bath temperature is higher than all likely laboratory ambient temperature. The times which should elapse between the two samplings at various temperatures for 5 cm depth are given below:

Temperature (°C)	Time	
	Hours	Minutes
10	5	14
12	4	56
14	4	40
16	4	26
18	4	13
20	4	10
22	3	49
24	3	39
26	3	30

A-5 RELATIVE DENSITY

A-5.1 Apparatus

A-5.1.1 Relative Density Bottle — 50 ml capacity, having a ground in capillary stopper.

A-5.1.2 Vacuum Pump — with a vacuum desiccator, capable of giving a vacuum of 10 mm.

A-5.2 Procedure

Weigh the empty, dry relative density bottle, introduce about 10 g of the material as dried in A-6 and weigh again. Nearly fill the relative density bottle with water and do not insert the stopper. Place it on a small tared evaporating dish and keep the assembly in the exhausted receiver of the vacuum pump overnight (at least 16 h). Fill up the relative density bottle carefully with water and insert the stopper. It is possible that a very small portion of the sample will run out of the sides of the stopper on inserting it. Wash down the particles thus forced out into the tared dish. Wipe off the excess of water from the relative density bottle, bring it to 27°C and weigh.

A-5.2.1 Evaporate the contents of the tared dish on a boiling waterbath under cover, dry to constant mass at 105 to 110°C.

A-5.2.2 Empty the bottle, fill it with water and weigh after bringing it to 27°C.

A-5.3 Calculation

$$\text{Relative density at } 27^{\circ}/27^{\circ}\text{C} = \frac{M_2 - M_1 - M_4}{(M_5 - M_1) - (M_3 - M_2) - M_4}$$

where

M_1 = mass in g of the empty relative density bottle,

M_2 = mass in g of relative density bottle and sample,

M_3 = mass in g of relative density bottle sample and water,

M_4 = mass in g of residue in tared dish, and

M_5 = mass in g of relative density bottle and water.

A-6 LOSS ON DRYING

A-6.1 Procedure

Weigh accurately about 20 g of the material and heat in a squat weighing bottle at 105 to 110°C. Cool and weigh till constant mass is obtained. Preserve the sample in a desiccator for subsequent tests.

A-6.2 Calculation

$$\text{Loss on drying, percent by mass} = \frac{100 M}{M_1}$$

where

M = Loss in mass in g, and

M_1 = mass in g of the material taken for the test.

A-7 LOSS ON IGNITION

A-7.1 Procedure

Weigh accurately about 1 g of the material in a porcelain or silica crucible. Ignite at about 1 000°C. Cool and weigh till constant mass is obtained.

A-7.2 Calculation

$$\text{Loss on ignition, percent by mass} = \frac{100 M}{M_1}$$

where

M = loss in mass in g, and

M_1 = mass in g of the material taken for the test.

A-8 MATTER SOLUBLE IN WATER

A-8.1 Reagents

A-8.1.1 Rectified Spirit — See IS 323 : 1959.

A-8.1.2 Bromophenol Blue Indicator Solution

Dissolve 0.1 g of bromophenol blue in 100 ml of rectified spirit.

A-8.1.3 Dilute Hydrochloric Acid — approximately 0.1 N.

A-8.2 Procedure

Weigh accurately about 10 g of the material in a 250-ml beaker. Add 5 ml of rectified spirit to wet the sample thoroughly. Add 200 ml of water, boil the suspension for 5 minutes and allow to cool to room temperature. Add sufficient bromophenol blue indicator to give a visible colour and then add dilute hydrochloric acid until the blue colour disappears.

NOTE — Bromophenol blue turns yellow at about pH 4 at which point flocculation occurs and clear filtration is obtained.

A-8.2.1 Transfer the contents of the beaker to a 250-ml volumetric flask, dilute to the mark with water and mix well by shaking. Filter through a filter paper, rejecting the first 50 ml of filtrate. Place 100 ml of the clear filtrate into a tared porcelain dish and evaporate to dryness on a water-bath. Dry the residue at $105 \pm 2^{\circ}\text{C}$, cool and weigh till constant mass is obtained.

A-8.3 Calculation

$$\text{Matter soluble in water, percent by mass} = 250 \times \frac{M_1}{M}$$

where

M_1 = mass in g of the residue obtained, and

M = mass in g of the material taken for the test.

A-9 MATTER SOLUBLE IN HYDROCHLORIC ACID

A-9.1 Reagents

A-9.1.1 Dilute Hydrochloric Acid — approximately 0.2 N.

A-9.2 Procedure

Weigh accurately about 2 g of the material. Transfer to a round-bottom flask with 100 ml of hydrochloric acid. Fix up a reflux condenser and boil the material on a sand bath for 5 minutes. Cool and filter through a sintered glass crucible No. G 4, Wash free from chlorides and evaporate the filtrate to dryness on a water bath in a silica basin. After gentle heating cool and weigh to constant mass.

A-9.3 Calculation

$$\text{Mass soluble in hydrochloric acid, percent by mass} = \frac{100 M}{M_1}$$

where

M = mass in g of residue, and

M_1 = mass in g of the material taken for the test.

A-10 COPPER**A-10.0 Outline of the Method**

Copper is determined colorimetrically using sodium diethyldithiocarbamate by visual comparison.

A-10.1 Apparatus

A-10.1.1 Nessler Cylinders — 100 ml capacity (see IS 4161 : 1967).

A-10.2 Reagents

A-10.2.1 Concentrated Ammonium Hydroxide — 18 (relative density 0.90).

A-10.2.2 Ammonium Citrate Solution

Dissolve 100 g of citric acid in 100 ml of ammonium hydroxide and make up to 200 ml with water.

A-10.2.3 Gum Arabic Solution — 5 percent (m/v).

A-10.2.4 Sodium Diethyldithiocarbamate Solution

Dissolve 1.0 g of sodium diethyldithiocarbamate in 1 000 ml of copper-free water. Keep in an amber colour bottle and protect from strong light.

A-10.2.5 Standard Copper Solution

Dissolve 0.3928 g of copper sulphate crystals ($\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$) in copper free water and make up the volume to 1 000 ml with copper free water. Take 100 ml of this solution and further dilute to 1 000 ml with copper-free water. One millilitre of this solution is equivalent to 0.01 mg of copper oxide (as CuO).

A-10.3 Procedure**A-10.3.1 Preparation of Sample Solution**

Weigh accurately 2.0 g of the dry sample and mix with 8 g of anhydrous sodium carbonate (see IS 296 : 1986), and fuse in a platinum crucible until the melt is clear. Leach out the melted mass with water into a porcelain dish, and then put the crucible in the same dish. Add about 20 ml of water and heat on a water-bath until the product is disintegrated. Remove the

crucible after washing in the dish. Evaporate the contents of the dish to dryness. Add a mixture of 12 ml of concentrated sulphuric acid (see IS 266 : 1993) and 2 ml of concentrated nitric acid (see IS 264 : 1976). Evaporate till fumes of sulphur trioxide appear, dilute to exactly 500 ml and filter through a filter paper to remove silica. Use this prepared sample solution for the test under A-13.

NOTE — In case of prepared sample solution is turbid, add a few drops of ferric chloride solution approximately 5 percent (m/v), boil and precipitate twice with ammonium hydroxide solution, and filter.

A-10.3.2 Pipette 25 ml of the prepared sample solution into a small conical flask or beaker. Drop into it a small piece of litmus paper and make the solution just alkaline with ammonium hydroxide. Add 2.5 ml of ammonium hydroxide in excess and heat to boiling. Allow to stand on a water-bath for one hour to ensure complete precipitation of aluminium hydroxide and then filter through filter paper (Whatman No. 1 or equivalent) into a Nessler cylinder, washing the filter paper with two or three small portions of hot water. To the solution in the Nessler cylinder add 5 ml of ammonium citrate, 5 ml of gum arabic solution, 10 ml of ammonium hydroxide solution and 10 ml of sodium diethyldithiocarbamate solution in the order mentioned. Dilute to 100-mark and mix well. To the other Nessler cylinder containing an equal aliquot of blank solution carried through the entire analysis in the same manner as the prepared sample solutions, and equal amounts of the same reagents, dilute to about 90 ml and mix. Add to this solution standard copper solution from a 10-ml burette until its colour matches that of the material under test after diluting to the same volume. Mix well after each addition of standard copper solution.

A-10.3.2.1 If the colour produced with the prepared sample solution is too deep for comparison, a smaller aliquot of the prepared sample solution from the acid digestion should be used.

A-10.4 Calculation

$$\text{Copper (as CuO), percent by mass} = \frac{0.02 V}{M}$$

where

V = volume in ml of standard copper solution used for the blank, and

M = mass in g of the dry material taken for the test.

A-11 ARSENIC

A-11.1 Procedure

Take 1.0 g of the dry material, disperse in 10 ml of water and add 5 ml of concentrated hydrochloric acid (see IS 265 : 1993) and 1 ml of stannous chloride solution. Carry out the test for arsenic as prescribed in IS 2088 : 1983, using for comparison 0.01 mg of arsenic trioxide (as As_2O_3).

A-12 IRON

A-12.0 Outline of the Method

Iron is determined volumetrically using stannous chloride solution and titrating the ferrous iron with standard potassium dichromate solution.

A-12.1 Reagents

A-12.1.1 Concentrated Hydrochloric Acid — See IS 265 : 1993.

A-12.1.2 Stannous Chloride Solution

Dissolve 5.0 g of stannous chloride crystals ($\text{SnCl}_2 \cdot 2\text{H}_2\text{O}$) in 60 ml of concentrated hydrochloric acid and dilute with water to 100 ml. Keep this solution in a stoppered bottle.

A-12.1.3 Mercuric Chloride Solution — saturated.

A-12.1.4 Concentrated Sulphuric Acid — See IS 266 : 1993.

A-12.1.5 Diphenylamine Sulphonate Indicator Solution

Dissolve 1.0 g of sodium or barium salt of diphenylamine sulphonate in 100 ml of concentrated sulphuric acid.

A-12.1.6 Standard Potassium Dichromate Solution — 0.05 N.

A-12.1.7 Phosphoric Acid — 85 percent (m/m).

A-12.2 Procedure

Weigh accurately about 10 g of the dry material and boil with 10 ml of hydrochloric acid and 30 ml of water for a few minutes. Filter and wash with water and discard the residue. Reduce the iron by adding stannous chloride solution until the yellow colour of ferric chloride disappears. Do not add more than 2 to 3 drops of stannous chloride in excess. Cool the solution, add rapidly mercuric chloride solution and stir so that a small white precipitate of mercurous chloride forms slowly, indicating that a slight excess of stannous chloride is present. Allow to stand for a minute or two, dilute to 200 ml, add 1 to 2 ml of phosphoric acid and 5 drops of diphenylamine indicator.

Titrate with standard potassium dichromate solution until the colour of the solution changes to a deep blue which does not fade on stirring.

A-12.3 Calculation

$$\text{Iron (as } \text{Fe}_2\text{O}_3 \text{), percent by mass} = 7.984 \times \frac{V N}{M}$$

where

V = volume in ml of standard potassium dichromate required for the titration,

N = normality of standard potassium dichromate solution, and

M = mass in g of the dry material taken for the test.

NOTE — For material with smaller iron content colorimetric method may be used for the determination of iron as prescribed in IS 3205 : 1984.

A-13 MANGANESE

A-13.0 Outline of the Method

Manganese is determined by comparing the colour produced by oxidation with periodate against the colour of a standard potassium permanganate solution.

A-13.1 Apparatus

A-13.1.1 Nessler Cylinders — 50 ml capacity (see IS 4161:1967).

A-13.2 Reagents

A-13.2.1 Phosphoric Acid — 85 percent (m/m).

A-13.2.2 Standard Potassium Permanganate Solution

Prepare a dilute solution of potassium permanganate by diluting to 50 ml in a volumetric flask a known volume of recently standardized solution in accordance with the following formula:

$$A = \frac{0.3524}{N}$$

where

A = volume in ml of the standard potassium permanganate solution to be made up to a volume of 50 ml, and

N = normality of the standard potassium permanganate solution.

One millilitre of this solution is equivalent to 0.1 mg of manganese oxide (as MnO). The solution shall be freshly prepared.

A-13.2.3 Potassium Periodate — solid.**A-13.3 Procedure**

Transfer 125 ml of the *prepared sample solution* (see A-10.3.1) to a 250-ml beaker and evaporate to 75 ml. Add 10 ml of the phosphoric acid, sprinkle 0.5 g of potassium periodate into the solution and heat to boil. Cool slightly, sprinkle again 0.1 g of potassium periodate and boil. When the colour appears to have developed to the maximum, place the beaker on a steam bath and keep for 15 minutes. If there is any doubt about the completeness of the reaction, add more potassium periodate. After the sample has stood on the steam bath for 15 minutes, remove the beaker and cool. Place the solution in a Nessler cylinder and dilute to mark with water. Place an equal aliquot of a blank solution carried through the entire analysis in the same manner as the *prepared sample solution* in the other Nessler cylinder and dilute almost to the mark. To this solution add standard potassium permanganate solution from a 10-ml burette until its colour matches that of the material under test when diluted to the same volume.

A-13.4 Calculation

$$\frac{\text{Manganese (as MnO),}}{\text{percent by mass}} = \frac{0.04 V}{M}$$

where

V = volume in ml of standard potassium permanganate solution used for the blank, and

M = mass in g of the dry sample taken for the test (see A-10.3.1).

A-14 pH VALUE**A-14.1 Procedure**

Weigh about 25 g of the material and transfer it to a 500-ml beaker. Add 100 ml of freshly boiled and cooled water. Allow to stand for 30 minutes with occasional stirring. Filter, reject the first 50 ml of the filtrate and collect the remaining filtrate in a beaker. Determine the pH of the solution by means of a suitable pH meter using glass electrodes.

A-15 OIL ABSORPTION**A-15.1 Procedure**

Place about 2 g of dry material, accurately weighed, on a glazed porcelain, ground glass or marble plate. Add linseed oil from a weighed dropping bottle, drop by drop and regularly, each drop being mixed well with the material using a palette knife. Incorporate the oil thoroughly in the course of 20 minutes, into the whole of the material with the palette knife, until a coherent mass is obtained. Weigh the dropping bottle again and determine by difference the weight of oil in grams. Where an approved sample is used for comparison, the oil absorption of the approved sample shall be determined by the same person and at the same time.

A-15.2 Calculation

$$\frac{\text{Oil absorption,}}{\text{ml per 100 g}} = \frac{100 M}{M_1}$$

where

M = mass in g of the linseed oil absorbed, and

M_1 = mass in g of the dry material taken for the test.

A-16 COLOUR (REFLECTANCE TO BLUE LIGHT OF WAVE-LENGTH 5040 Å)**A-16.1 Outline of the Method**

The colour of the sample is compared with that of Lovibond magnesium oxide block.

A-16.2 Procedure

By means of a suitable arrangement, light falling on Lovibond magnesium oxide block shall be reflected on a selenium cell connected to a galvanometer. The galvanometric deflection is proportional to light reflected. For comparison, the reading obtained by magnesium oxide block is taken as 100.

A-16.2.1 Substitute the magnesium oxide block with a block prepared from a sample of the material. Measure the reflectance and report as colour (or reflectance) compared to that of magnesium oxide as 100.

ANNEX B

(Clause 6)

SAMPLING OF LIGHT KAOLIN

B-1 GENERAL REQUIREMENTS OF SAMPLING

B-1.0 In drawing, preparing, storing and handling test samples, the following precautions and directions shall be observed.

B-1.1 Samples shall not be taken in an exposed place.

B-1.2 Sampling instrument shall be clean and dry when used.

B-1.3 Precautions shall be taken to protect the samples, the material being sampled, the sampling instrument and the containers for samples from adventitious contamination.

B-1.4 The samples shall be placed in clean, dry air-tight glass or other suitable containers.

B-1.5 The sample containers shall be of such a size that they are almost completely filled by the sample.

B-1.6 Each sample container shall be sealed air tight with a stopper after filling and marked with full details of sample, the date of sampling and other identification particulars.

B-1.7 The samples shall be stored in such a manner that during storage the properties of the material do not get effected.

B-1.8 The sampling shall be done by a person or persons mutually agreed to between the purchaser and the supplier.

B-2 SCALE OF SAMPLING FOR MATERIAL PACKED IN BAGS**B-2.1 Lot**

In a single consignment all the material belonging to the same grade and coming from the same supplier or source shall constitute a lot.

B-2.2 Sub-lot

For the purpose of sampling all the material in a lot shall be divided into a suitable number of homogeneous sub-lots of approximately equal weight in accordance with col 1 and 2 of Table 2.

B-2.3 The number of bags to be sampled from each sub-lot is given in col 3 of Table 2. These bags shall be selected at random from the sub-lot. In order to ensure the randomness of selection, random number tables shall be used.

For guidance and use of random number tables, IS 4905 : 1968 may be referred. In the absence

of random number tables, following procedure may be adopted.

Starting from any bag in the lot, count them as 1,2,3,....., etc, up to r and so on. Every r th container thus counted shall be withdrawn, r being the integral part of N/n , where N is the total number of containers in the lot, and n the number of containers to be chosen.

Table 2 Scale of Sampling for Bags

(Clauses B-2.2 and B-2.3)

No. of Bags in the Lot	No. of Sub-Lots	No. of Bags to be Sampled from each Sub-Lot
(1)	(2)	(3)
Up to 10	1	All
11 to 200	2	5
201 to 500	3	10
501 to 2 000	4	15
2 001 and above	5	20

B-2.4 From each of the bags selected, a small but approximately equal portion of material shall be taken. All the portion of material taken from the sample bags in the same sub-lot shall be mixed together to constitute a composite sample so that there are as many composite samples as the number of sub-lots into which a lot has been divided.

B-3 SAMPLING FROM WAGONS**B-3.1 Lot**

In a single consignment all the material belonging to the same grade and coming from the same supplier or source shall constitute a lot.

B-3.2 Sub-Lot

For the purpose of sampling all the wagons in a lot shall be divided into a suitable number of homogeneous sub-lots of approximately equal weight in accordance with Table 3.

Table 3 Scale of Sampling for Wagons

Weight of the Material, tonnes	No. of Sub-Lots	No. of Increments from each Sub-Lot
(1)	(2)	(3)
Up to 200	2	20
201 to 500	3	30
501 to 2 000	4	40
2 001 and above	5	50

B-3.3 A minimum of 25 percent of wagons shall be selected at random from the sub-lot. The corresponding number of increments to be taken from a sub-lot shall be distributed over the selected wagons with a view to determining the number of increments that should be taken from each of the selected wagons in the sub-lot.

B-3.4 All the increments taken from the selected wagons in a sub-lot shall be pooled together to constitute a gross sample.

B-4 PREPARATION OF SAMPLES

B-4.1 All the increments from the same sub-lot shall be thoroughly mixed to constitute a gross sample representing the sub-lot. The minimum size of the composite sample/gross sample shall be about 3 kg. If the gross sample is less than this, additional number of increments/portions shall be drawn so as to make up the required quantity.

B-4.2 The composite sample/gross sample shall be suitably reduced by the method of coning and quartering or any other suitable method to about 750 g which shall be divided into three portions called laboratory sample, one for the purchaser, another for the supplier and the third as the referee sample, each duly placed in the sample container.

B-5 NUMBER OF TESTS AND CRITERIA FOR CONFORMITY

B-5.1 One laboratory sample from each sub-lot shall be tested individually for all the requirements of this specification and the sub-lot shall be considered satisfactory if the laboratory sample satisfies all the requirements of this specification.

B-5.2 The lot shall be declared to conform to the requirements of the specification if all the sub-lots (*see B-5.1*) are found satisfactory.

ANNEX C

(Foreword)

COMMITTEE COMPOSITION

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Composition of Alums and Phosphates Subcommittee, CHD 03 : 01

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BUREAU OF INDIAN STANDARDS

Headquarters:

Manak Bhavan, 9 Bahadur Shah Zafar Marg, New Delhi 110002

Telephones : 331 01 31, 331 13 75

Telegrams : Manaksanstha
(Common to all Offices)

Regional Offices :

Central : Manak Bhavan, 9 Bahadur Shah Zafar Marg
NEW DELHI 110002

Telephone

{ 331 01 31
{ 331 13 75

Eastern : 1/14 C. I. T. Scheme VIII M, V. I. P. Road, Maniktola
CALCUTTA 700054

{ 37 84 99, 37 85 61
{ 37 86 26, 37 86 62

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{ 60 38 43,
{ 60 20 25,

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